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(19)

(54) METHOD OF AND APPARATUS FOR EFFECTING INTERACTION BETWEEN LIQUID AND SOLID

(71) We, NORTHERN ENGINEERING INDUSTRIES LIMITED, a British Company of NEI House, Regent Centre, Newcastle upon Tyne NE3 3SB do hereby declare the invention for which we pray that a patent may be granted to us, and the method by which it is to be performed to be particularly described in and by the following statement:—

The invention relates to apparatus for, and methods of, perfoming ion exchange between cellulosic ion exchange medium and liquid containing proteinaceous materials.

Typically, such liquid is effluent from food and drink processing plants such as milk whey from milk processing plants for example. The effluent may be treated either to recover useful proteinaceous material; or merely to render the effluent liquid less noxious and suitable for discharge to a river or for re-cycling in some other way.

In this specification the term "proteinaceous material" accordingly means protein and related substances and enzymes and related substances which are amenable to ion exchange processes with cellulosic ion exchange media.

UK Patent No. 1 436 547 and UK Patent No. 1 387 265 describe such processes and methods of making cellulosic media for use in such processes. So far, however, the practical application of such processes is at a very early stage in its development and no large-scale plants have yet operated in any country.

Apparatus for performing ion exchange between cellulosic ion exchange medium and liquid containing proteinaceous material, according to the invention, comprises a vessel containing a filter means extending at least partly across the vessel and agitator means above the filter means operable to mix the medium and the liquid during ion exchange, the vessel having inlet means above the filter means and outlet means below the filter means.

In one form of apparatus for performing the method according to the invention using more than one vessel, each vessel contains a filter means extending at least partly across the vessel and agitator means above the filter means operable to mix the medium and the liquid during ion exchange, each vessel having inlet means above the filter means and outlet means below the filter means and each of the vessels having an exit port above and adjacent to the filter means, the apparatus also comprising receptacle means, one for each said vessel to receive output from the respective exit port thereof, each vessel being connected to a respective outlet of one of the receptacle means and the apparatus also comprising tank means having outlets connected to respective inlet means of at least some of the vessels and each vessel being connected to a source of differential air pressure.

In another form of apparatus for performing the method according to the invention using more than one vessel, each vessel contains apparatus for performing the method claimed in claim 1 using more than one vessel, each vessel containing a filter means extending at least partly across the vessel and agitator means above the filter means operable to mix the medium and the liquid during ion exchange, each vessel having inlet means above the filter means and outlet means below the filter means, the apparatus also comprising tank means having outlets connected to respective inlet means of at least some of the vessels, the vessels having their outlet means connected to conduits leading to respective tank means, and each vessel being connected to a source of differential air pressure.

A method of performing ion exchange between cellulosic ion exchange medium and liquid containing proteinaceous material comprising using at least one vessel contain-

ing filter means below an agitator means and having inlet and outlet means respectively above and below the filter means, the method also comprising:—

(a) mixing the medium with a volume of said liquid in one said vessel by operating the agitator means to load the medium with material and to deplete said first volume of liquid;

(b) separating loaded medium from said depleted first volume in one said vessel by applying differential air pressure across said filter means therein and aiding passage of said depleted volume therethrough by said applied differential air pressure there across, said first volume being passed out of said vessel;

(c) adding wash liquid to the medium and operating the agitator means to mix the wash and medium and to wash the latter;

(d) separating the medium from the wash liquid in one said vessel by applying differential air pressure across said filter means and aiding passage of wash liquid therethrough by said applied differential air pressure thereacross, said wash liquid being passed out of said vessel;

(e) adding regenerant to the medium and operating the agitator means to mix the regenerant and medium and to strip proteinaceous material from the medium and so regenerate it and to load the regenerant with proteinaceous material;

(f) separating the regenerated medium from the loaded regenerant in one said vessel by applying the differential air pressure across said filter means and aiding passage of loaded regenerant therethrough by said applied differential air pressure thereacross, said loaded regenerant being passed out of said vessel;

(g) repeating steps (a) to (f) with subsequent volumes of said liquid.

Examples of apparatus and of ways of performing the method will now be described to illustrate the invention with reference to the accompanying drawings in which:—

Figure 1 is a schematic view of apparatus in the form of a typical reactor vessel for use in the method with part cut away to show the interior construction;

Figure 2 is a schematic diagram in the form of a flow-sheet showing a typical form of apparatus and the method;

Figures 3(a) to 3(g) are schematic diagrams showing successive stages of operation in the method practiced using the apparatus shown in Figure 2;

Figure 4 is a schematic diagram of yet another form of apparatus shown as a flow sheet according to which the method can be performed;

Figures 5 and 6 are respectively a schematic diagram of another form of apparatus shown as a flow sheet according to which the method can be performed and a sequence chart of stages and events in that flow sheet form of the method; and

Figure 7 is a schematic diagram of yet another form of apparatus shown as a flow sheet according to which the method can be performed.

Figure 1 shows an upright cylindrical vessel 10 which is constructed to withstand internal pressures of up to two atmospheres though in use it is unlikely that normal pressure greater than about one half atmosphere would be necessary. The vessel is made of stainless steel or reinforced synthetic plastic material, for example, and is typically suitable for handling milk whey as the liquid to be treated by interaction with a particulate cellulosic ion exchange medium, for example. The vessel 10 contains an agitator 12 which may be rotatable by a shaft 14; or else the agitator 12 may be a vibratory agitator capable of vertical vibration by a vibratory drive mechanism (not shown) connected to the upper end of the shaft 14. The shaft 14 is rotatable by a motor mounted on top of the vessel 10.

The vessel 10 also contains a filter means 16 which may be a wedge-wire screen; a metallic grid; a filter cloth; or a perforate metal plate, though other forms of filter means may be used. The means 16 has apertures of a size, shape and profile such as to allow efficient separation of liquid from the ion exchange medium without blinding or blocking of the apertures. The means 16 extends entirely across the full diameter of the vessel 10. As shown, the filter means 16 consists of a retaining ring 100, a gasket 102 of material, such as Neoprene; a sheet 104 of filter wire mesh material of grade IN 316; a support grid 106; a second sheet 108 of filter wire mesh material of grade IN 316; a 1/4 inch thick plate 110 drilled with 1/2 inch diameter holes at spacing of several inches; and another Neoprene gasket 112 resting on an inner flange 114. The filter means 16 is secured together by bolts 116.

The vessel 10 has an upper inlet and/or outlet means 18 controlled by valves (not shown) for liquid and air feed; the vessel has an air vent not shown leading to atmosphere. The vessel 10 has a lower outlet 30 for filtrate beneath the filter 16.

Where necessary, the vessel has another outlet just above the filter means 16 and controlled by a valve for ion exchange medium (not shown) (see later description of other systems).

The vessel 10 has a Ph probe 118.

Figure 2 shows a system in which a reactor vessel 10 is used and which consists of a bulk milk whey storage tank 200 connected by a pump 202 and a valve 204 to the vessel 10; a water feed pump 206; an air supply line 208; a hold tank 210 having a lower outlet connected via a valve 212 to the upper end of the vessel 10 and to a pump 214. The pump 214 feeds a product tank 216.

The base of the vessel 10 has an outlet 30 (Figure 1) leading to a valve 218 which has two outlets; one outlet is connected via a re-cycle pump 220 to the upper end of the vessel 10. The same outlet is connected to valves 222 and 224. The latter is connected to the upper end of the hold tank 210. An air supply line 226 is connected to the upper end of the hold tank 210.

An acid make-up tank 228 is connected via an acid dosing pump 230 to the vessel 10; and an alkali make-up tank 232 is connected via a pump 234 to the vessel 10.

The system shown in Figure 2 is capable of operating according to a "1 : 1: 1" mode that is, providing one stage of loading of the medium with proteinaceous material; one stage of washing of the medium with water; and one stage of regeneration of the medium to remove proteinaceous material from the medium in solution in the regenerant liquid.

Figures 3(a) to 3(g) show clearly the full cycle of events.

Figures 3(a) shows whey coming in from the tank 200. The cellulosic material is represented as quiescent at 201. Figure 3(b) shows pH correction using hydrochloric acid from the tank 228 and absorption of proteinaceous material by the cellulosic medium as the medium and whey are agitated and mixed by the agitator 12.

The cellulosic medium becomes loaded with proteinaceous material from the whey and agitation is ceased, after which air is applied under pressure above the slurry of depleted whey and loaded medium as shown at Figure 3(c) to assist drainage of depleted whey liquid through the filter means 16. The depleted liquid passes to drain. The medium is thus separated from liquid after which clean water is admitted to the vessel 10 from a source not shown. Then the agitator 12 is operated to ensure mixing of medium and water to ensure washing of the medium as shown in Figure 3(d).

Air is again admitted under pressure to assist separation of medium and wash water (this stage is not shown) the wash water passing to drain or to a separate water storage tank (not shown).

Next, air under pressure is passed into the hold tank 210 to force water contained therein into the vessel 10 (Figure 3(e)). After this, caustic soda solution is added to give a regenerant liquid of the correct pH and the agitator 12 is operated to cause thorough mixing of medium with the regenerant solution (Figure 3(f)). The medium loses proteinaceous material and is regenerated. The regenerant becomes enriched with proteinaceous material.

Air is then admitted under pressure to the vessel 10 to assist separation of the regenerant from the medium. The air pressure forces regenerant through the filter means 16 and out of the vessel 10. The regenerant or product liquor as it is called goes either direct to an ultra filtration plant for separation of useful proteinaceous material or to the hold tank 210.

The option of returning the product to the tank 210 is shown only for the sake of completeness to indicate that, if desired, a second stage of adsorption may be effected by mixing the product liquor with completely regenerated medium in the vessel 10 so as to further enrich the product liquor.

Such a mode would be a 2.1.1 mode. If desired two stages of regeneration can be used so that a 2.1.2 mode is practiced.

The fully enriched liquor would be passed to the tank 210 then immediately pumped from the tank 210 to the product hold tank 216 by the pump 214, the valve 212 being operated accordingly

In Figures 3(b), 3(d) and 3(f) the pump 220 is shown in operation to circulate liquid below the filter means 16 back into the vessel above the filter means 16. This is to ensure optimum treatment of liquid during takeup of protein by the medium; to ensure optimum water usage during washing; and to ensure optimum enrichment of the liquor during regeneration of the medium. The vessel shown is of the type having a full dished base for strength to resist the internal pressure. It is also possible to use a flat bottomed vesel in which the filter means is close to the base provided the base is made strong enough. In that case the volume of liquid beneath the filter means is small and forced re-circulation is not necessary. Natural circulation through the filter means ensures optimum liquid/medium interaction in such cases.

All the stages are performed in the vessel 10, the medium remaining in the vessel throughout. This is an especially simple system with the advantage of absence of transfer of medium from one vessel to another thus reducing power requirements

The system shown in Figures 2 and 3 based on the vessel shown in Figure 1 represents the simplest single module type of system. The differential air pressure across the filter means 16 and throughput requirements give a basic vessel size of some 8 feet diameter by 8 feet height of vessel .Such a basic module is capable of handling some 10,000 gallons per day of milk whey, for

example. A typical plant may require 6 modules giving a capacity of 60,000 gallons per day.

A wash following the stage of loading of the medium with protein is essential to wash out materials not taken up by the medium. These are typically lactose and crude proteinaceous material. Also, the adhering crude liquid is washed away at this stage.

In a modification (not shown) the filter means may extend across only part of the base of the vessel; for example it may extend over only a central portion. It may take the form of a low upright annular filter screen cylinder closed at its upper end by a flat horizontal filter screen wall. The cylinder would extend above the general level of the base of the vessel by only several inches or not more than about a foot. The vessel base could be generally flat in such a case.

Figure 4 shows a reactor vessel 250; containing an agitator 251; a filter means 252; liquid level controls or load cells 253; and a probe 254 for monitoring the pH value of liquid in the vessel. The vessel has at its upper end an inlet 260 controlled by a valve 262 for raw liquid feed; an inlet 264 controlled by a valve 266 for pressurised air; an inlet 268 controlled by valve 270 for wash water and inlet 272 controlled by valve 274 for addition of acid 300 reagent; an inlet 276 controlled by valve 278 for addition of alkali 302 reagent and an inlet 280 controlled by valve 282 for desorbent regenerant addition.

The vessel has an upper outlet valve 284 for air pressure release after each liquid discharge stage.

The vessel has a lower outlet 286 with valve 288 for recycled wash water; a valve 290 for discharge to drain; and a valve 292 for recycle of desorbent eluate i.e. protein-rich regenerant.

Tanks 293, 294 and 295 are the respective hold tanks.

All the stages are performed in the vessel 250. The sequence of operations is as follows, assuming the vessels 250 to contain a particulate cellulosic ion exchange medium in a condition of maximum receptivity ready for take-up of protein from milk whey.

Raw liquid milk whey is fed from the tank 293 to the vessel 250 by pump 400 to bring the level up to the top level control device or the load decided by load cells. Concurrent with this charging acid is added to the whey in reactor 250 to give the correct ion exchange pH of 3.

The agitator 251 is operated to mix the liquid and medium at the correct pH condition, so that protein in the liquid is taken up by the medium, which thus becomes laden with protein. This stage is the first stage of treatment of the milk whey effluent stream. The liquid in the vessel 250 now contains a considerably reduced quantity of true potein and can be passed to drain via the outlet 286 and the valve 290, or to an intermediate tank (not shown) should more than one stage of adsorption be required.

Pressurised air is passed into the vessel 250 via the valve 266 and inlet 264 to accelerate filtering of the liquid from the medium.

Next, water is passed into the vessel 250 from the tank 294 via the pump 402, the valve 270 and inlet 268. This water is previously used water stored in the tank 294 so as to reduce total water usage. The agitator 251 is operated to mix the medium and water so as to cause unwanted substances such as lactose to be diluted and washed out of the medium. The water is drained away via the outlet 286 and valve 290; some water can be returned to the tank 294 via the line 416 and the valve 288. Air pressure is applied above the liquid surface in the vessel 250 to force the water into the tank. Excess water is passed to drain via the valve 290.

Additional wash stages can be effected if desired until the medium is brought to a desired condition, these wash stages being direct or countercurrent.

Next, partly-spent regenerant solution or desorbent is passed into the vessel 250 from the tank 295 via the pump 403 the line 404, the valve 282 and the inlet 280. The agitator 251 is operated to mix the medium and regenerant so as to complete a first stage of regeneration of the medium. The regenerant takes protein into solution and so becomes spent. The medium becomes fully regenerated, having lost its protein content. The spent regenerant is passed to the tank 295 via the outlet 286, line 418 and the valve 292, with applied air pressure providing the moving force on the liquid surface in the vessel 250.

The tank has an outlet 450 by which protein rich regenerant liquor is passed to subsequent stages in the overall process. After removal of some liquor from the tank 295 fresh caustic soda solution is added to the tank 295 from a source (not shown) so as to restore the concentration of regenerant in the liquid in the tank 295. That is, the regenerant then contains relatively little protein.

The medium is thus ready to interact with raw feed material from the tank 293 as described above at the beginning of the cycle, which can be repeated using the same ion exchange medium. Ultimately, the medium becomes less effective and partly degraded. It may be necessary to remove the medium entirely from the vessel 250 which can be effected via an outlet 452 and valve 454.

Fresh medium can be passed into the

vessel 250 through an upper opening (not shown) having a lid, or as a slurry by means of pump (not shown) from a tank (not shown).

A typical operating sequence for the method performed by the system shown in Figure 4 would be:—

1. Mix raw liquor with medium 15 - 20 minutes;
2. Drain liquor off — 5 to 10 minutes;
3. Introduce wash water 5 minutes;
4. Mix to wash medium 10 minutes;
5. Drain wash water off 5 to 10 minutes;
6. Introduce regenerant i.e. de-sorbent liquid — 5 minutes. Also, add alkali to give pH=9;
7. Mix to cause de-sorbent to take up proteinaceous material — 15 to 25 minutess;
8. Drain de-sorbent off — 5 to 10 minutes;
9. Introduce raw milk whey liquor — 5 minutes at pH=6. Introduce acid simultaneously to give pH=3.

A typical cycle would take about 3 hours. About 3 hours per day are needed for full stripping of the medium using caustic soda solution to ensure perfect hygiene.

This is known as "cleaning in place". Thus, some 7 cycles per day are possible.

Figure 5 shows a third embodiment of apparatus and method in which five reactor vessels R1 to R5 are used, each similar to the vessel 10 described above. Six hold tanks HT1 to HT6 are provided with three pumps P1 to P3 associated, respectively, with the outlets from the tanks HT1; HT2 and HT3. The pumps are arranged to feed liquid from the tanks to upper inlets of the reactor vessels via an array of pipelines and control valves CV. Outlets at the bases of the reactor vessels are connected via further lines to the upper inlets of the tanks HT1 - HT4 via control valves CV and manually operable valves V.

A water feed line 320 is provided and also a raw effluent feed line 322 via a pump 324 both leading to the upper inlets of the reactor vessels.

The tank HT6 has a water feed line 326 and water level control devices 328, 330. The outlet from the tank HT6 is connected to the upstream side of the pump P1.

The tank HT1 has an overflow outlet to the tank HT5, from the base outlet of which protein-rich liquid can be drawn at 331.

The tank HT4 has its base outlet connected to a drain connection 332.

In Figure 5 CV denotes a control valve operable automatically and V denotes a manually operable valve which can be used in emergencies, and during setting-up procedures. Air supply lines, and acid and alkali pH correction supplies have been omitted, as have the fresh regenerant supply and a complete system of water re-cycling, for simplicity.

The method carried out in each tank is similar to that described in relation to Figure 4 and it is sufficient to refer to Figure 6 which gives a complete cycle of operation.

In Figure 6 it must be noted that the liquid in HT1 is partly passed to HT5 and the remaining liquid in HT1 has fresh regenerant such as caustic soda solution added before liquid is drawn from HT1 to fill any reactor vessel. As required, water from HT6 may also be drawn off to complement the feed to any vessel from HT1.

The tank HT1 always contains a solution of protein in regenerant liquid, the protein content being either maximum or minimum. The tank HT2 always contains partly treated liquid feed. The tank HT3 always contains partly spent regenerant. The tank HT4 always contains fully treated liquid effluent and is intermittently partly emptied to drain via line 332.

The pumps P1, P2 and P3 are operable to feed liquid from the tanks HT1, HT2 and HT3 to the vessels R1, R2, R3 and R4 as required. Air pressure applied above liquid in the vessel is used to force liquid from the vessels to the tanks HT1 - HT4.

Figure 7 shows yet another embodiment of the invention in which five reactor vessels R1 to R5 are used, together with two hold tanks HT1 and HT2 and five transfer cones C1 to C5.

In this embodiment the medium is moved from vessel to vessel in counter-flow to the direction of transfer of liquid being treated. In Figure 7 solid lines represent pipelines along which liquid flows from hold tanks to vessels and vice-versa; broken lines represent pipe-lines along which slurry of medium and liquid flows from vessels to cones and vice-versa; and chain dotted lines represent pipelines along which pressurised air is supplied to vessels to force liquid out of them or through which air is vented from vessels.

If one particular batch of feed is first of all considered it is shown as being passed via the valve V6 into the vessel R1 where it is mixed by the operation of the agitator with partly spent medium so as to fully exhaust the medium (i.e. to cause it to hold a maximum of protein). The protein content of the input liquid is thus partly reduced (i.e. the first stage of protein removal has been effected).

Next, the slurry of medium and liquid is forced by air pressure to C3 and almost immediately passed from C3 to R3. The fully spent medium fully loaded with protein remains in R3. The liquid passes out of R3 via V15 under applied air pressure to hold tank HT1. The liquid next flows from HT1 into R2 where it is mixed with fully regenerated (i.e. protein free) medium so that the second stage of removal of pro-

tein from the liquid is effected. Air is vented from R2 via the chain dotted line to the vent valve V24 during flow into R2.

Next, the slurry of medium and liquid is forced by air pressure into the cone C1 and almost immediately flows into R1. Next, the valve V13 opens and fully treated liquid as effluent passes to drain while pressurised air is applied over the slurry in the vessel to assist filtration. The partly spent medium, carrying protein but not fully loaded remains, being retained by the filter means in R1.

The fully loaded medium which was left in R3, being retained by the filter means therein, is washed by agitation with water during the reaction stage simultaneously with the second stage of protein removal from the liquid in R2. Next, the slurry of water and medium is transferred by air pressure to C4 and almost immediately to R4 where it is agitated further. Next, the valve V16 opens to allow wash water to flow to drain under applied air pressure, while the fully loaded medium remains in R4 retained by the screen means.

Next. partly spent regenerant (i.e. containing some dissolved protein) is passed from HT2 into R4 and agitated with the medium to exhaust the regenerant fully (i.e. to bring the protein content to maximum). The first stage of protein removal from the medium is thus effected the medium being partly regenerated and now holds a lesser protein content. Next, the slurry of medium and liquid is forced by air pressure to C5 and thence to R5.

The valve V17 opens to allow protein rich regenerant liquor to pass to a further tank 500, under applied air pressure to assist filtration.

The tank 500 has a liquid level contol 501 for fresh regenerant agent such as caustic soda solution and has an offtake 502 so that protein rich liquor can be drawn off for subsequent treatment. Then fresh caustic soda solution is run in to restore the protein concentration to a minimum.

Next, the fresh regenerant is passed into R5 from the tank 300 and mixed with the medium so as to effect the second stage of regeneration of the medium, i.e. the second stage of protein removal.

Next, the slurry is transferred to C2 via valve V22 and thence to R2. The valve V14 opens to allow partly spent regenerant to flow to HT2, under applied air pressue.

The medium is thus fully regenerated.

The partly spent regenerant liquor contains the last of the protein from the batch of input liquid first considered. It is passed from HT2 to R4 where it is mixed with medium carrying protein from the next batch of input liquid to be treated. Ultimately, the regenerant liquor now fully loaded with protein passes with the medium under ai rpressure to C5 and thence to R5. The liquor then passes from the medium via V17 to the product tank 500, under applied air pressure.

That completes the removal of protein from an input batch of liquid to be treated.

Figure 7 shows three overall stages:

STAGE I: in which interactions occur namely: first stage of protein take-up by ion exchange between raw input liquid partly-spent medium in R1; second stage protein take-up by ion exchange between partly-treated liquid and fully-regenerated medium in R2; washing of medium in R3; first stage of protein removal from medium in R4 by ion exchange with regenerant liquid; and second stage protein removal from medium by ion exchange with regenerant liquid in R5.

Status

1. Cones C1 C2 C3 C4 C5—empty
2. Liquor hold tanks HT1 HT2—charged
3. Eluant (Regenerant) feed system—charged

Actions

1. All valves shut
2. Valves V6 V7 V8 V9 V10 V24 open
3. Level controls on reactors R1 R3 R5 will define start of reaction period
4. Agitators started
5. Operation takes places for reaction period
6. All valves shut.

The wash stage removes unwanted lactose and other soluble pollutants and loose particles from the medium. The method described is a 2:1:2 method.

STAGE II: in which medium slurry is transferred from cones to vessels and from vessels to cones. It comprises two phases:

Phase A

Status

1. All reaction products in reactors R1 R2 R3 R4 R5
2. Liquor hold tanks HT1 HT2 empty

Actions

1. All valves shut
2. Valves V18 V19 V20 V21 V22 V23 opened
3. Agitators rotating
4. After full transfer (P of air line pressure) all valves shut.

Phase B

Status

1. All reaction products in C1 C2 C3 C4 C5
2. Liquor hold tanks HT1 HT2 empty
3. Reactors R1 R2 R3 R4 R5 empty

Actions

1. All valves shut
2. V24 opened
3. Valves V1 V2 V3 V4 V5 opened
4. Agitators rotating
5. After full discharge—all valves shut.

STAGE III: in which liquid is transferred from vessels, some to hold tanks, and from hold tanks to vessels:

Status

1. All reaction products in reactors R1 R2 R3 R4 R5
2. Transfer cones C1 C2 C3 C4 C5 empty

Actions

1. All valves shut
2. Valves V13 V14 V15 V16 V17 opened
3. V23 opened
4. Agitators stopped
5. After full transfer (P of line pressure) all valves shut.

In Figure 7 the hold tanks are higher than the vessels so that flow is by gravity from the tanks.

handling of the medium is such as to minimise degradation by attrition and pressure. The medium is either not moved at all from the vessel in which it interacts with liquid; or else it is transferred using airpressure to cones in which the medium has but a brief residence. Little or no settling occurs in the cones and the medium slurry is very quickly passed on to the next vessel and agitated once more to maintain its optimum physical characteristic. This is especially important with cellulosic ion exchange medium, full particulars of which and its preparation for use may be had from THE VISCOSE DEVELOPMENT COMPANY LIMITED, ENGLAND who supply suitable medium under their registered trademark "VISTEC'. Both anion and cation type medium is available and although the methods described above are especially intended for recovery of milk whey protein, using anion medium, the methods generally are applicable for treatment of many liquid streams, whether for clean-up only to render the effluent suitable for discharge to rivers etc. or also for recovery of valuable organic materials contained in the liquid such as proteins, enzymes, and other macromolecular substances.

The filter-bottom reactor vessel described above has been subjected to tests and found satisfactory throughout 2000 cycles of operating using 50 mesh woven wire cloth. Alternatively, a wedge wire screen as mentioned above may be used; or some other kind of filter means.

The further treatment of the protein-rich liquid drawn off from the product tank as described above is not described in detail but it is sufficient to say that the solution is concentrated by ultra-filtration, or by direct evaporation, the latter process probably requiring the previous crystallisation-out of impurities before final evaporation and final drying. Ultra-filtration allows impurities to pass away from the protein; also washing of the product is feasible within the ultra-filtration unit if required.

The raw input liquid can be subjected to pre-treatment if necessary to remove coarse solid particles, fat or other ingredients which could reduce the efficiency of the ion exchange and washing stages.

The descriptions above have omitted description of the period cleansing actions which are required to ensure clean, hygenic conditions of operation so that protein recovered is safe and free from any harmful contamination. This precaution is typically necessary in plant handling milk whey liquid.

The method described with reference to Figure 6 has an added advantage that the medium is repeatedly being removed from the reactor vesels so that continuous monitoring or laboratory checking of the physical, chemical, biological and other properties of the medium is readily feasible. This means that very high safety standards can be maintained. Medium can readily be extracted from the cycle and replaced when necessary.

Using the methods described above large throughputs can be handled of say the order of 60,000 gallons per day.

The filter-bottom reactor vessels have been described as having air pressure applied above the slurry during filtering to assist drainage of liquid through the filter means. Vacuum-assisted filtration may be used instead of positive air pressure.

The methods described above by way of example can be modified to change the number of stages of protein uptake, protein removal from medium, or washing of medium. If necessary wash stages can be introduced to wash medium after regeneration too.

Many different operational paterns are possible all based on the use of one or more filter-bottom reactor vessels of the kind described, in which medium either remains throughout or between several of which medium is transferred. Alternatively, medium may remain in a vessel for more than one stage and then be moved to another vessel for subsequent stages.

In the methods described above, the medium is either separated from liquid in the same vessel in which interactions occur; or else the medium is subjected to separation from liquid before it is fully spent. In the method described with reference to Figure 7, the basic sequence is; agitate the slurry; transfer the slurry; filter to separate liquid from medium; and pass further liquid into the vessel to which

the slurry is transferred. An alternative method is feasible in which the sequence is: agitate the slurry; filter in the same vessel to separate liquid from medium; pass further liquid into the same vessel; and transfer the resulting slurry.

In the apparatus described above, the process control valves can be operated automatically under the control of a programmable master controller. Manually operable valves are also necessary as mentioned but have been omitted from Figure 7 for simplicity. Controls for liquid levels and for monitoring pH values have been mentioned and it is feasible to incorporate those and other controls which may be necessary into an automatically controlled system.

WHAT WE CLAIM IS:—

1. A method of performing ion exchange between cellulosic ion exchange medium and liquid containing proteinaceous material comprising using at least one vessel containing filter means below an agitator means and having inlet and outlet means respectively above and below the filter means, the method also comprising:

(a) mixing the medium with a volume of said liquid in one said vessel by operating the agitator means to load the medium with material and to deplete said first volume of liquid;

(b) separating loaded medium from from said depleted first volume in one said vessel by applying differential air pressure across said filter means therein and aiding passage of said depleted volume therethrough by said applied differential air pressure there across, said first volume being passed out of said vessel;

(c) adding wash liquid to the medium and operating the agitator means to mix the wash liquid and medium and to wash the latter;

(d) separating the medium from the wash liquid in one said vessel by applying differential air pressure across said filter means and aiding passage of wash liquid therethrough by said applied differential air pressure thereacross, said wash liquid being passed out of said vessel;

(e) adding regenerant to the medium and operating the agitator means to mix the regenerant and medium and to strip proteinaceous material from the medium and so regenerate it and to load the regenerant with proteinaceous material;

(f) separating the regenerated medium from the loaded regenerant in one said vessel by applying differential air pressure across said filter means and aiding passage of loaded regenerant therethrough by said applied differential air pressure thereacross, said loaded regenerant being passed out of said vessel;

(g) repeating steps (a) to (f) with subsequent volumes of said liquid.

2. A method according to claim 1, in which all of steps (a) to (g) are performed in each of one or more said vessels.

3. A method according to claim 2, in which a sequence of steps (a), (b), (a), (b) . . . . is performed before step (c) using at steps (a) respective volumes of liquid.

4. A method according to claim 2 or 3, in which a sequence of steps (c), (d), (c), (d) . . . . is performed before step (e) using at steps (c) respective volumes of wash liquid.

5. A method according to claim 2, 3 or 4, in which a sequence of steps (e), (f), (e), (f) . . . . is performed before step (g) using at steps (e) respective volumes of regenerant.

6. A method according to claim 1, in which more than one said vessel is used, and which comprises performing step (a) in a first of said vessels, transferring the mixed medium and said volume of liquid to a second of said vessels, performing steps (b) and (c) in said second vessel, transferring the mixed medium and wash liquid to a third of said vessels, perfoming steps (d) and (e) in said third vessel, transferring the mixed medium and regenerant to said first vessel and performing step (f).

7. A method according to claim 6, in which, prior to step (c), a sequence of steps (a), transfer to a subsequent one of said vessels, (b), (a), transfer to a subsequent one of said vessels, (b). . . . is performed using at steps (a) respective volumes of liquid, there being a total number of vessels sufficient to cater for the total number of steps in the sequence.

8. A method according to claims 6 or 7, in which prior to step (e), a sequence of steps (c) transfer to a subsequent one of said vessels, (d), (c), transfer to a subsequent one of said vessels, (d). . . . is performed using at steps (c) respective volumes of wash liquid, there being a total number of vessels sufficient to cater for the total number of steps in the sequence.

9. A method according to claim 6, 7 or 8, in which prior to step (g), a sequence of steps (e), transfer to a subsequent one of said vessels, (f), (e), transfer to a subsequent one of said vessels, (f). . . . is performed using at steps (e) respective volumes of regenerant, there being a total number of vessels sufficient to cater for

the total number of steps in the sequence.

10. A method according to any of claims 6 to 9, in which there occurs simultaneously at each of the respective vessels the steps of:

(i) a mixing operation as specified in steps (a), (c) and (e); or

(ii) transfer of medium and liquid out of the vessel; or

(iii) transfer of medium and liquid into the vessel; or

(iv) a separating operation as specified in steps (b), (d) and (f); or

(v) introduction of one of said liquids and regenerant into the vessel.

11. A method according to any of claims 1 to 10, in which the regenerant loaded with proteinaceous material is treated to recover the proteinaceous material which can be subjected to further purification treatment if required.

12. A method according to any of claims 1 to 11, in which, during steps (a), (c) and (e), liquid below the or each filter means is extratced from the or each vessel and re-introduced into the or each vessel above the or each filter means.

13. Apparatus for performing the method claimed in claim 1 using more than one vessel, each vessel containing a filter means extending at least partly across the vessel and agitator means above the filter means operable to mix the medium and the liquid during ion exchange, each vessel having inlet means above the filter means and outlet means below the filter means and each of the vessels having an exit port above and adjacent to the filter means, the apparatus also comprising receptacle means, one for each said vessel to receive output from the respective exit port thereof, each vessel being connected to a respective outlet of one of the receptacle means and the apparatus also comprising tank means having outlets connected to respective inlet means of at least some of the vessels and each vessel being connected to a source of differential air pressure.

14. Apparatus for performing the method claimed in claim 1, using more than one vessel, each vessel containing a filter means extending at least partly across the vessel and agitator means above the filter means operable to mix the medium and the liquid during ion exchange, each vessel having inlet means above the filter means and outlet means below the filter means, the apparatus also comprising tank means having outlets connected to respective inlet means of at least some of the vessels, the vessels having their outlet means connected to conduits leading to respective tank means, and each vessel being connected to a source of differential air pressure.

15. Apparatus according to any claim of claims 13 or 14, in which an outlet means of each vessel is connected to the inlet of a pump having an outlet connected to an inlet means of the vessel.

16. Apparatus according to any claim of claims 13 to 15, in which each filter means comprises a generally flat horizontal perforate screen.

17. Apparatus according to claim 16, in which the perforate screen comprises wire mesh.

18. Apparatus according to claim 16, in which the perforate screen comprises a wedge wire screen.

19. Apparatus for performing the method claimed in claim 1 substantially as hereinbefore described with reference to Figure 1 of the accompanying drawings.

20. Apparatus for performing the method claimed in claim 1 substantially as hereinbefore described with reference to Figures 1 and 7 of the accompanying drawings.

21. Apparatus for performing the method claimed in claim 1 substantially as hereinbefore described with reference to Figures 1, 2 and 3(a) to 3(g) of the accompanying drawings.

John Richardson from here on 1 559 809

22. Apparatus for performing the method claimed in claim 1 substantially as hereinbefore described with reference to Figures 1 and 4 of the accompanying drawings.

23. Apparatus for performing the method claimed in claim 1 substantially as hereinbefore described with reference to Figures 1, 5 and 6 of the accompanying drawings.

24. A method according to claim 1, substantially as hereinbefore described with reference to Figures 1, 2 and 3 of the accompanying drawings.

25. A method according to claim 1, substantially as hereinbefore described with reference to Figures 1 and 4 of the accompanying drawings.

26. A method according to claim 1, substantially as hereinbefore described with reference to Figures 1, 5 and 6 of the accompanying drawings.

27. A method according to claim 1, substantially as hereinbefore described with reference to Figures 1 and 7 of the accompanying drawings.

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1559809 COMPLETE SPECIFICATION

8 SHEETS *This drawing is a reproduction of the Original on a reduced scale*

Sheet 1

Fig.1.

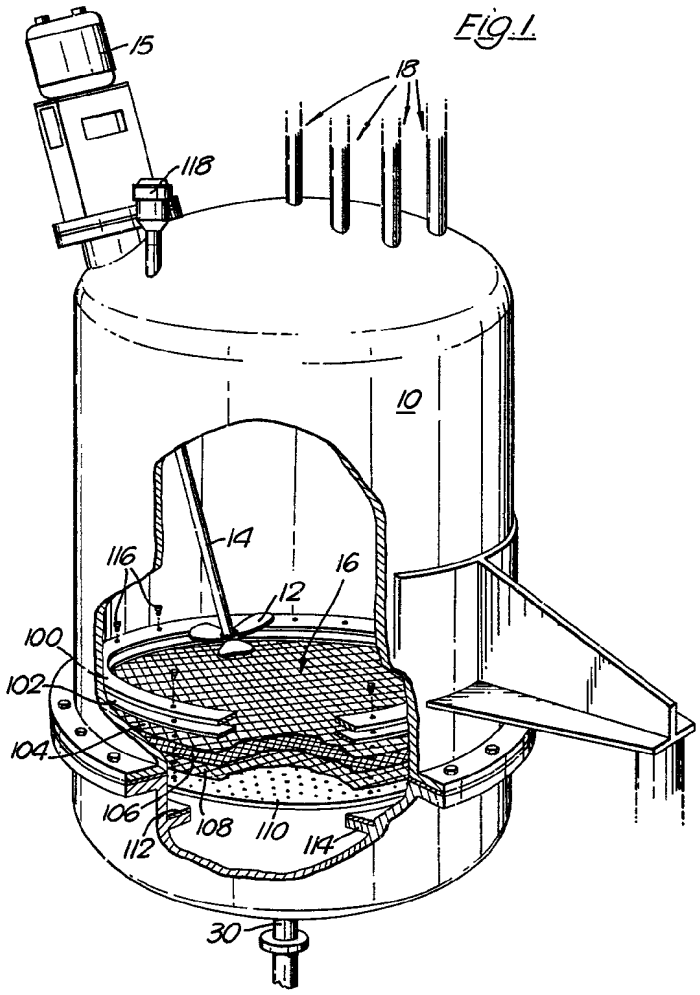

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Fig.2

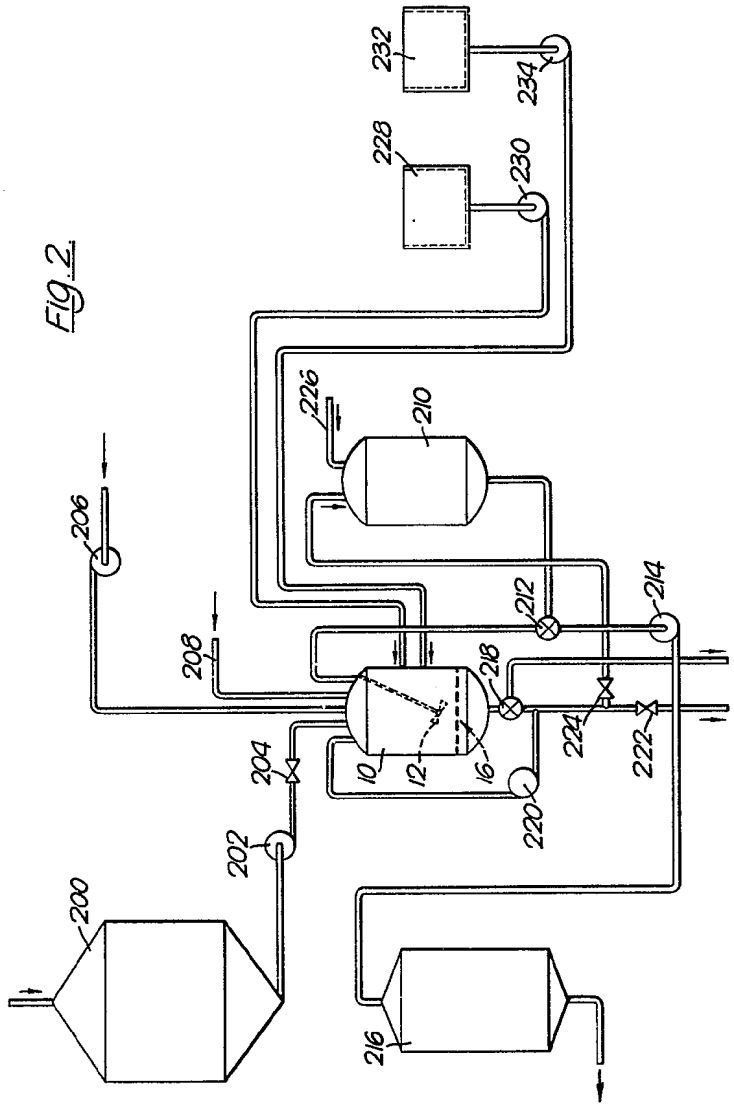

Fig.3a.

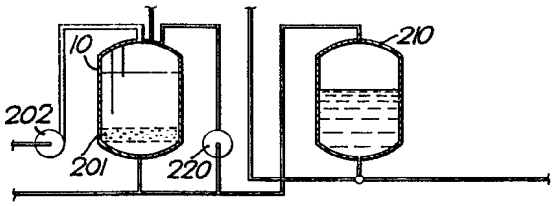

Fig.3b.

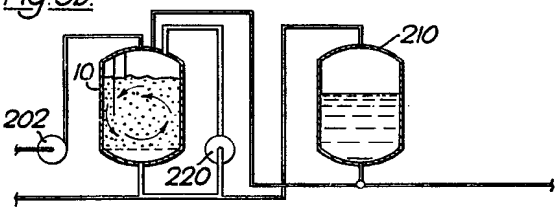

Fig.3c.

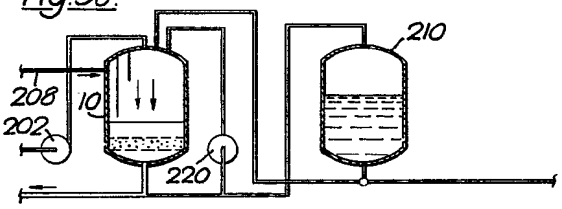

Fig.3d.

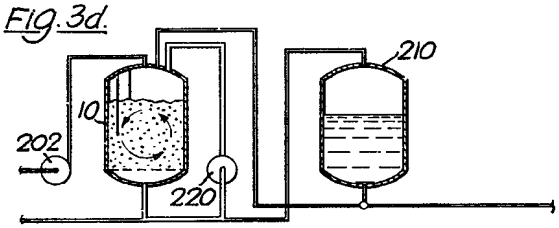

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Fig. 3e.

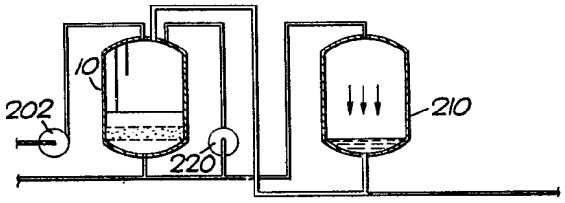


Fig. 3f.

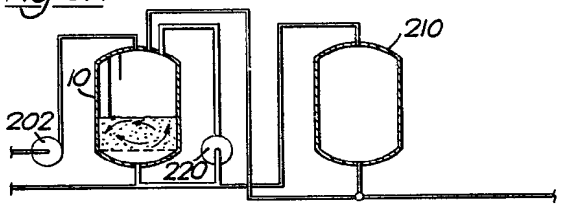
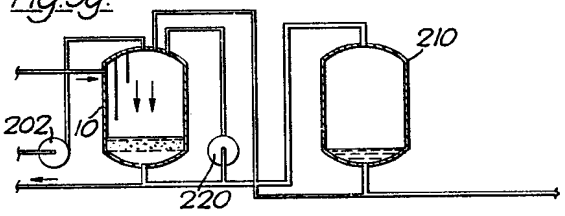


Fig. 3g.



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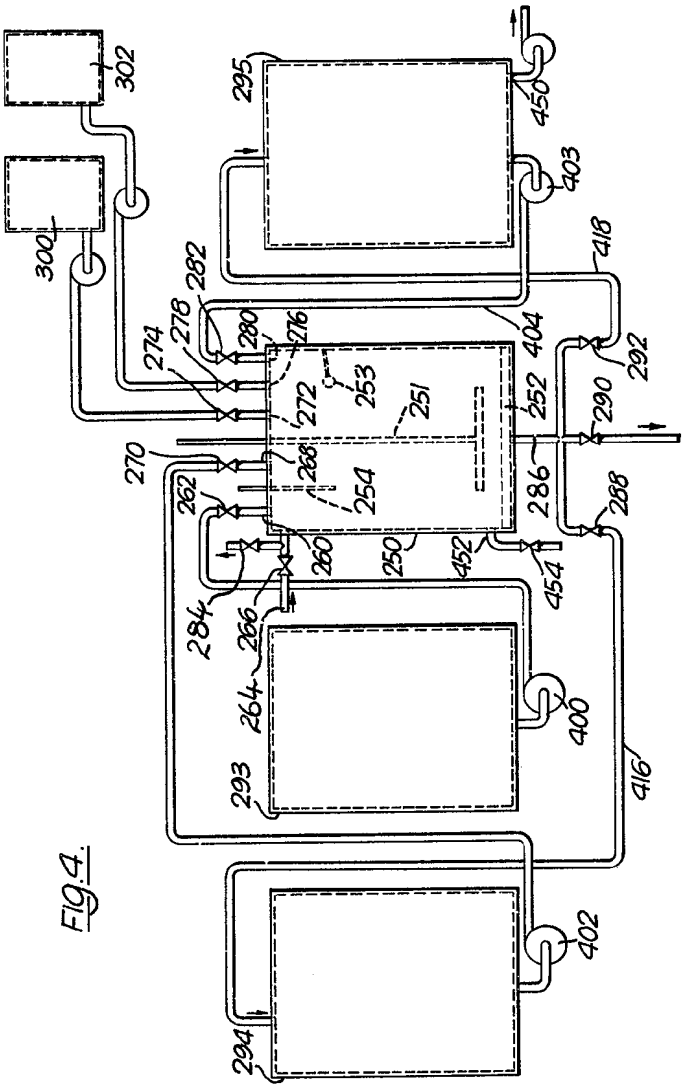

FIG.4.

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Fig.5.

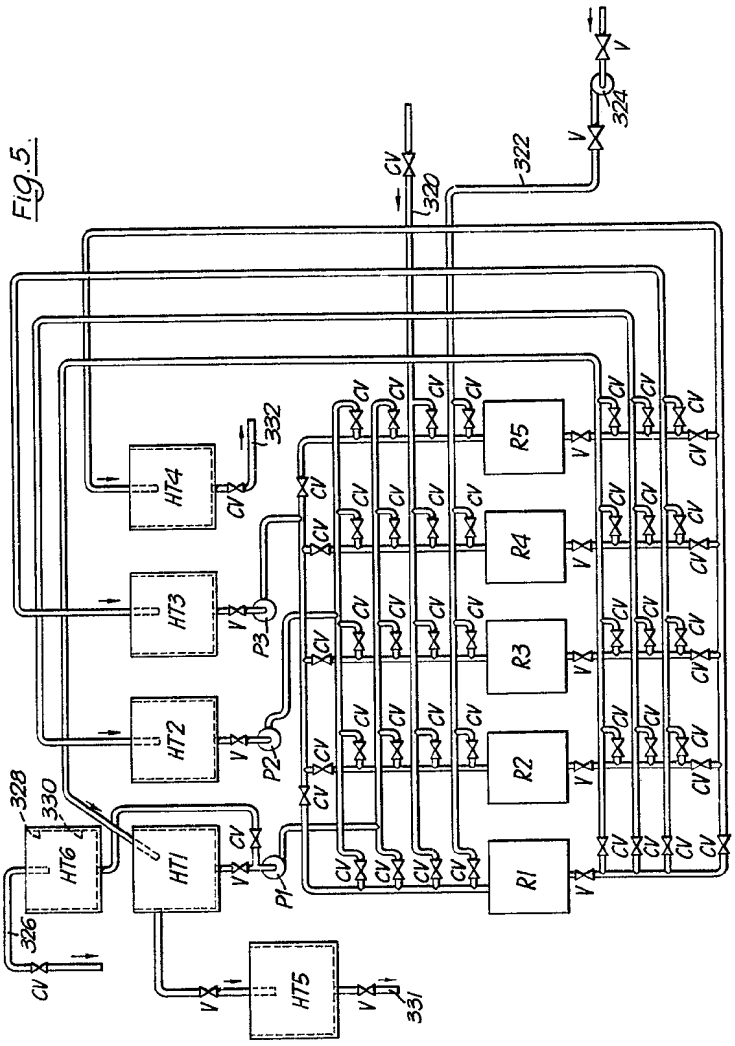

Fig.6.

MINUTES	R1	R2	R3	R4	R5
0	FILL FEED	FILL WASH WATER	FILL EX. HT3	FILL EX. HT1	FILL EX. HT2
10	1ST. STAGE PROTEIN REMOVAL FROM FEED	WASH	1ST. STAGE OF REGENERATION OF MEDIUM	2ND. STAGE OF REGENERATION OF MEDIUM	2ND. STAGE PROTEIN REMOVAL FROM FEED
20		DRAIN TO HT4		DRAIN TO HT3	
30	DRAIN TO HT2	FILL EX. HT3	DRAIN TO HT1	FILL EX. HT2	DRAIN TO HT4
40	FILL WASH WATER	1ST. STAGE OF REGENERATION OF MEDIUM	FILL EX. HT1	2ND. STAGE PROTEIN REMOVAL FROM FEED	FILL FEED
50	WASH		2ND. STAGE OF REGENERATION OF MEDIUM	DRAIN TO HT4	1ST. STAGE PROTEIN REMOVAL FROM FEED
60	DRAIN TO HT4	DRAIN TO HT1	DRAIN TO HT3	FILL FEED	DRAIN TO HT2
70	FILL EX. HT3	FILL EX. HT1	FILL EX. HT2	1ST. STAGE PROTEIN REMOVAL FROM FEED	FILL WASH WATER
80	1ST. STAGE OF REGENERATION OF MEDIUM	2ND. STAGE OF REGENERATION OF MEDIUM	2ND. STAGE PROTEIN REMOVAL FROM FEED		WASH
90		DRAIN TO HT3		DRAIN TO HT2	DRAIN TO HT4
100	DRAIN TO HT1	FILL EX. HT2	DRAIN TO HT4	FILL WASH WATER	FILL EX. HT3
110	FILL EX. HT1.	2ND. STAGE PROTEIN REMOVAL FROM FEED	FILL FEED	WASH	1ST. STAGE OF REGENERATION OF MEDIUM
120	2ND. STAGE OF REGENERATION OF MEDIUM	DRAIN TO HT4	1ST. STAGE PROTEIN REMOVAL FROM FEED	DRAIN TO HT4	
130	DRAIN TO HT3	FILL FEED	DRAIN TO HT2	FILL EX. HT3	DRAIN TO HT1
140	FILL EX. HT2	1ST. STAGE PROTEIN REMOVAL FROM FEED	FILL WASH WATER	1ST. STAGE OF REGENERATION OF MEDIUM	FILL EX. HT1
150	2ND. STAGE PROTEIN REMOVAL FROM FEED		WASH		2ND. STAGE OF REGENERATION OF MEDIUM
160	DRAIN TO HT4	DRAIN TO HT2	DRAIN TO HT4	DRAIN TO HT1	DRAIN TO HT3

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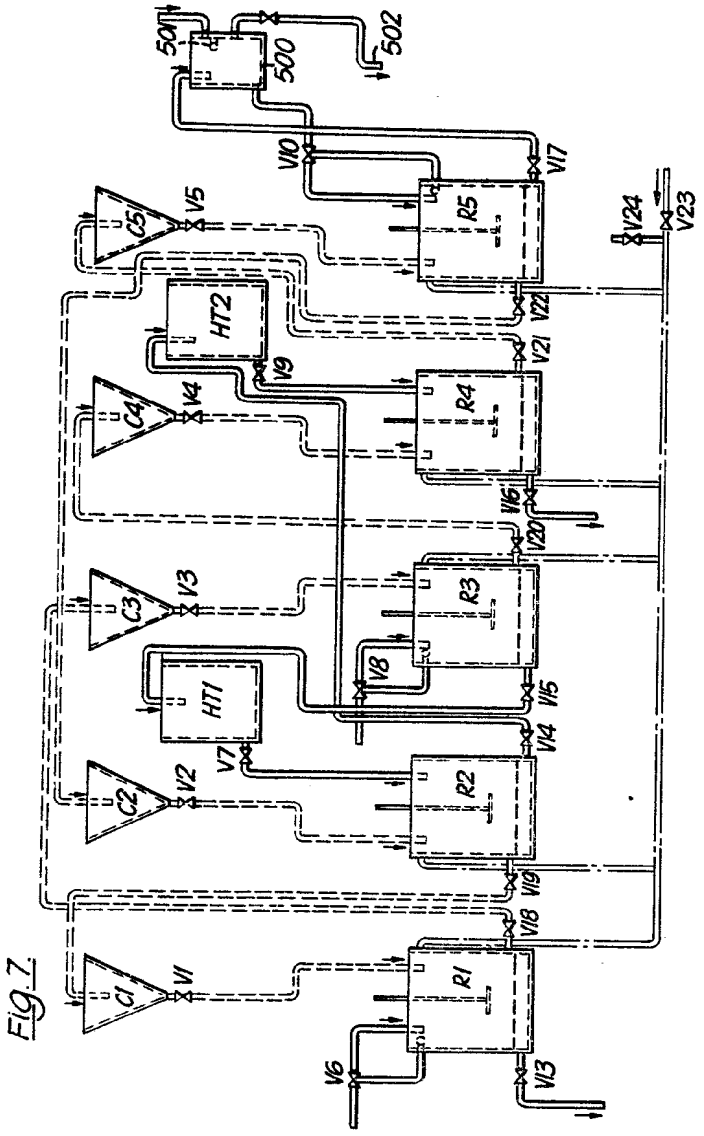

Fig.7